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TITLE:
SUBMICRON, UNBACKED, SHAPED METAL FOILS

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Submicron, Unbacked, Shaped Metal Foils

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Implosion experiments often utilize cylindrical, thin metal foils. Previously these foils were made either from flat sheets, with a seam where the edges joined, or in the form of a composite polymer/metal laminate, with the plastic film acting as a supporting substrate.

A method has been developed to produce unbacked, shaped metal foils in sub-micron thicknesses. This process utilizes a temporary substrate consisting of a water-soluble polymer film as a base for the electron-beam deposition of the metal layer. After formation of the metal foil, the polymer is removed by immersion of the assembly in water. Unbacked metal-foil cylinders as thin as 0.17 μm with extremely smooth, wrinkle-free surfaces have been produced by this technique.

1. Background

Cylindrical thin metal foils are of interest to a number of laboratories conducting pulsed power experiments.[1] The earliest work in this area was carried out using commercially available flat foils. These were simply wrapped around a pair of support rings to give a foil cylinder.[2] The seam at the point where the edges of the foil were joined was highly undesirable from the standpoint of implosion symmetry. It presented a region of essentially zero mass if a butt joint was formed, or an area with twice the mass of the main body of the foil when an overlap joint was utilized.

To overcome the problems associated with this joint, a technique for producing seamless foils was developed by Kindel, Reinovsky and Clark.[3] Their process utilized a cylindrical polymer substrate made according to the method of Grader.[4] The product was not a pure metal foil, but rather a composite consisting of a polymer film substrate with a continuous and uniform metal overlay. While this seamless polymer/metal laminate was an improvement over the previous wrapped foils, it presented a number of problems in fabrication and use. The polymer film was formed from a noxious organic solvent by a dip-casting process. Because the polymer was vaporized in the experiment along with its metal coating, non-uniformity in the polymer film contributed to asymmetries in the implosion process. Thus great care was required to produce a polymer film cylinder with uniform thickness, both from top to bottom and around its perimeter. The polymer film was originally formed on the inner surface of a stainless steel ring. For metalization and subsequent experimental use, it had to be transferred to another fixture. This step involved inserting the metalization fixture inside the forming ring with a clearance of only about 0.25 mm between the two parts. This close tolerance led to frequent tearing of the thin polymer film during the transfer process.

Because of its laminate nature, wrinkles were invariably present in the polymer/metal composite. These were undesirable, of course, from the standpoint of implosion symmetry. Finally, the vaporization of the polymer film consumed a significant fraction of the energy available in the implosion process, but contributed nothing towards the goal of the experiments. Thus it was necessary to keep the mass density of the polymer substrate as low as possible. This in turn led to the production of very thin films (about 1 micron thick) which only exacerbated the handling problems described above.

The developments reported herein arose directly from attempts to improve and simplify the process for producing thin cylindrical foils. As a result of this work, unbacked thin metal foils with mass densities in the range of 50 $\mu g/cm^2$ have been produced. These foils are extremely smooth and virtually wrinkle-free.

2. Experimental Procedure

The method of Kindel et. al.[2] was the basis for these experiments, but the technique was greatly modified A polymer film substrate was formed by dipping a stainless steel ring into a solution consisting of 9.8% polyvinyl alcohol, 2.4% glycerin and 87.8% water. The polyvinyl alcohol was 88% hydrolyzed and had an average molecular weight of about 110,000. The rate of immersion of the forming ring and its removal from the casting solution was not closely controlled. The excess liquid was allowed to drip away from the forming ring before placing it in a forced air oven at 70°C for 3-4 hours to evaporate the solvent. After this time, the polymer film could be readily pulled away from the interior surface of the ring as illustrated in Fig. 1. In some cases, the forming ring had been pre-treated with a commercial mold release spray (Mold Release 225, RAM Chemical, Gardena, Ca.) to facilitate removal of the polymer film from the ring, but this was not an essential step.

After removal from the forming ring the polymer film was pulled over the ring electrode fixture as shown in Fig. 2. This could be done easily since the forming ring was sized to give a cylindrical polymer film larger in diameter than the electrode fixture by 0.50 mm. The polymer film was attached to the outer edge of each ring electrode by using a cotton swab wet with water to dissolve away the excess polyvinyl alcohol film, while at the same time utilizing a portion of the dissolved polymer as a glue to hold the film to the electrodes (see Fig. 3).

The film/electrode assembly was then immersed in isopropyl alcohol for 30 seconds to extract a portion of the glycerin plasticizer and subsequently dried for 10 minutes at 70°C. The effect of this treatment was to cause the film to shrink tightly around the electrode fixture. Figure 4 shows a comparison of a film after this treatment, on the left, with a mounted but untreated film on the right.

The fixture, with the polymer film suspended tautly across the electrodes, was mounted in a rotator for vacuum deposition of the metal foil. The metal was applied from an electron beam source. In all cases, the coating assembly was designed to confine the angle of incidence of the metal vapor to the film to greater than about 60° from the horizontal, since previous work had shown that low angles of incidence have a tendency to produce films with poor physical properties.[5] When aluminum foils were produced, a short pulse of oxygen gas was intermittently admitted to the vacuum system to produce a partial oxide layer at about every 0.1 μm of aluminum deposition. This procedure has been reported to increase the mechanical strength of the aluminum foil significantly.[6]

In the final step in the process, the metalized film/foil composite was lowered into a water bath at a rate of 0.5 mm/min. to dissolve away the polymer backing. Careful attention was required at this step to prevent rupture of the metal foil as its polymer backing was being dissolved away. The temperature of the water bath was maintained at 35-45°C. Two drops of liquid dishwashing detergent were added to lower the surface tension, and insure uniform wetting of all surfaces. After immersion of the fixture to about the middle of the upper ring electrode, the water was slowly drained away through a drip valve at a rate of about 0.5 ml/min. The foil, now unbacked, was allowed to air dry before being placed in a closed container for storage prior to use.

3. Discussion

Initially, attempts were made to produce unbacked metal foils simply by dissolving away the polyvinyl formal substrates from composite polymer/metal laminates made by the procedure of Kindel et. al.[2] All attempts to dissolve the polymer backing were unsuccessful, however.

Since the polymer film was formed from a solution, and polyvinyl formal is known to be soluble in a large number of organic solvents,[7] it was concluded that the polymer must have cross-linked during the metalization process under the effects of heat and/or radiation.

A search was then begun for a different polymer substrate. The criteria for candidate polymers included the following: 1) The material was required to be an inherently good film former to give films with the strength and flexibility needed to withstand the handling involved in the fabrication, transfer, and metalization processes. 2) The polymer film had to release readily from the forming ring without tearing or damage to the surface of the film. 3) The film had to exhibit excellent surface properties, being free of bubbles, flecks, and other imperfections which would tend to replicate in the metal layer deposited on it. 4) Finally, the polymer film had to be readily soluble after metalization. Examination of available literature indicated that polyvinyl alcohol could readily meet the first three criteria.[8] Experiments proved that it could also fulfill the last criterion. The fact that polyvinyl alcohol is water soluble greatly simplified the preparation of the polymer films since no noxious vapors were released at any stage of the process. In addition, since polyvinyl alcohol is non-toxic, waste materials could be disposed of by simply flushing them down the drain.

Because of the extremely good release characteristics of the polyvinyl alcohol film, it could be stripped away by hand from the stainless steel forming ring to give a free-standing polymer cylinder.

This cylinder could be readily pulled over the electrode fixture with the aid of a pair of tweezers. Since this film was intended for removal prior to implosion of the metal foil, the mass and thickness uniformity of the film were of little importance. It was advantageous, in fact, to prepare a film approximately 25 μm thick to assure that it would not tear during handling. The manual transfer of the film from the forming ring to the electrode fixture eliminated the close tolerances which had been a problem in the earlier process.

Once the polyvinyl alcohol film was placed over the electrode fixture and glued to the ring electrodes, it was necessary to draw it tightly across the gap between them in order to give a smooth surface for metalization. After some experimentation, it was found that this could be accomplished by incorporating glycerin into the forming solution. The glycerin acts as a plasticizer for the polyvinyl alcohol film. It thus helps assure flexibility of the film during handling. More importantly it is soluble in isopropyl alcohol, and is extracted upon immersion of the film in this solvent. The resultant loss of mass causes the film to shrink tightly around the electrodes producing the smooth surface required for metalization.

The metal deposition was confined to a high angle of incidence and gas pulsing was utilized when aluminum was deposited in order to obtain metal foils with the highest possible strengths.[5,6] This was particularly important because the metal had to be self-supporting across the 2 cm electrode gap after removal of the polymer substrate.

The polyvinyl alcohol substrate rapidly dissolved as the metalized film was lowered into the water in the extraction step. This was evidenced by the periodic appearance of a shock wave which traveled around the circumference of the foil at the surface of the water. Examination of the back surface of a foil by Auger Electron Analysis showed a residual carbon layer of less than 0.05 μm. This was attributed to surface contamination.

Figure 5 shows a typical aluminum/polyvinyl formal laminate foil. This foil has a layer of aluminum about 0.2 μm thick on its surface, but a mass density in excess of 100 $\mu g/cm^2$ because of the presence of the polymer backing. Note the wrinkles in the surface of the foil. The presence of these wrinkles is not surprising since thin layers of two dissimilar materials must conform to each other in this structure. The coefficient of thermal expansion of aluminum, for example, is on the order of 10 times greater than that of polyvinyl formal. Figure 6 is a photo of an unbacked aluminum foil made as described herein. This foil is 0.2 μm thick with a mass density of 54 $\mu g/cm^2$. There is no extraneous contribution to the mass because there is no supporting substrate. The surface is seen to be shiny and virtually wrinkle free, since the aluminum need not conform to a second material. Gold foils as thin as 0.20 μm have also been made by this process.

4. Conclusions

This work has demonstrated that sub-micron, unbacked. thin metal foils can be produced by electron beam vapor deposition. Polyvinyl alcohol provides an excellent substrate for the deposition process. It is readily removed by water after fabrication of the foil. As a result of this development, higher quality foils can be obtained with lower mass densities than were previously available. Implosion experiments can be conducted with lower levels of energy input and more efficient energy utilization.

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Figure Captions

Figure 1

The Polyvinyl alcohol film is easily removed from stainless steel forming ring.

Figure 2

Film cylinder fits loosely over electrode fixture.

Figure 3

Water is used to dissolve excess polymer and tack film to outer edges of ring electrodes.

Figure 4

Extraction of glycerin plasticizer causes film to shrink tightly around ring electrodes (left). Prior to extraction, film is loose and has many wrinkles (right).

Figure 5

Composite aluminum/polyvinyl formal foil. Note wrinkles.

Figure 6

Unbacked aluminum foil. Surface is smooth and shiny.

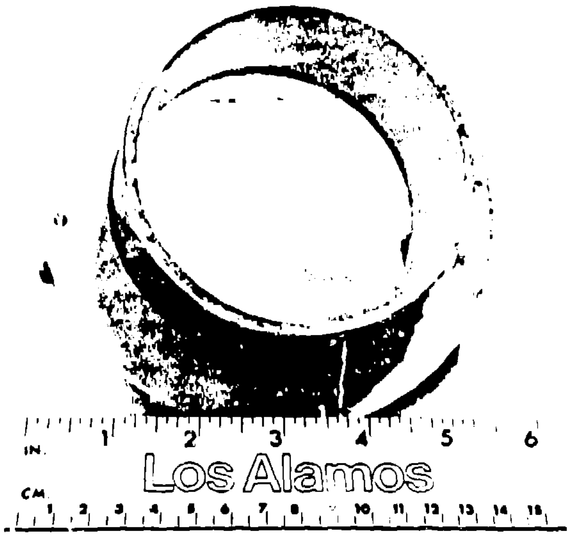
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Fig. 1

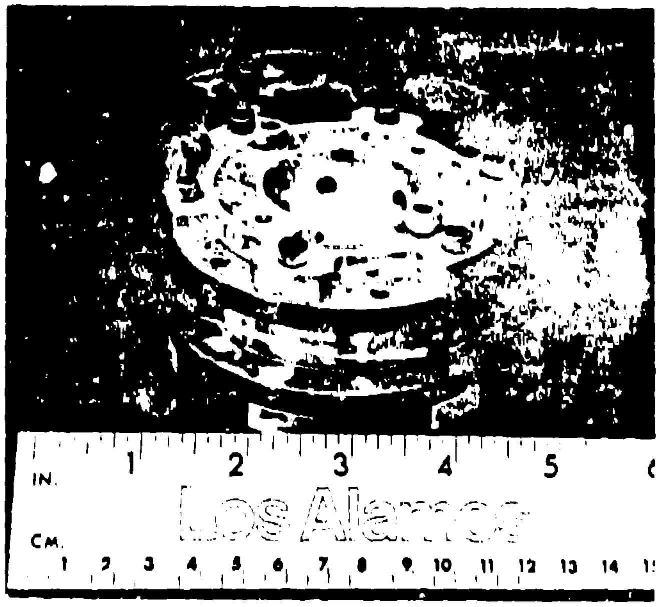
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Fig. 2

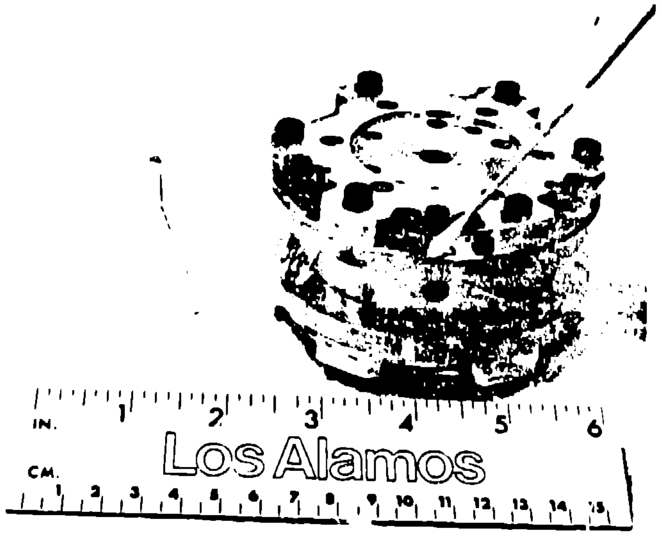
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Fig. 3

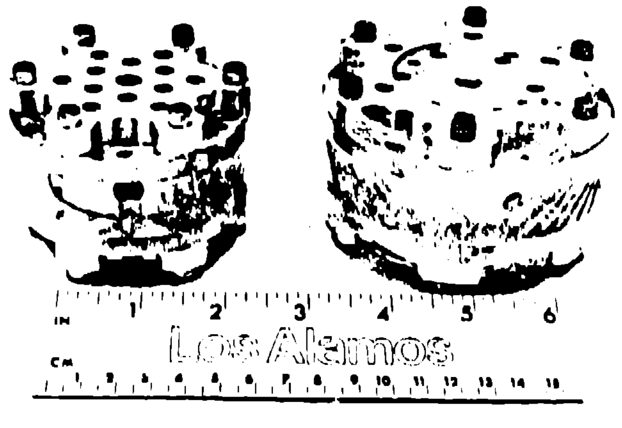
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Fig. 4

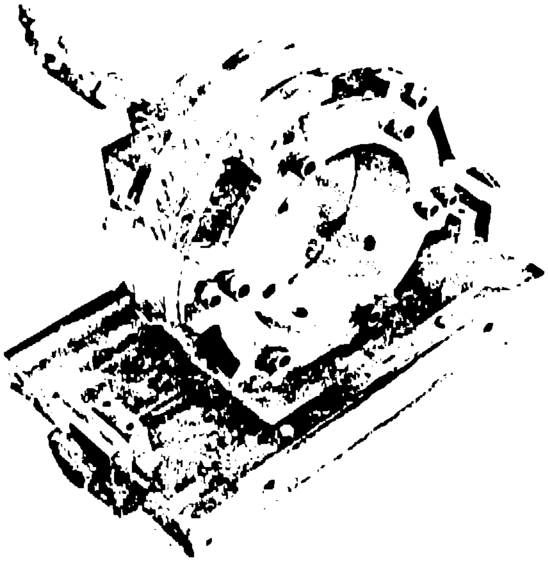

Fig. 5

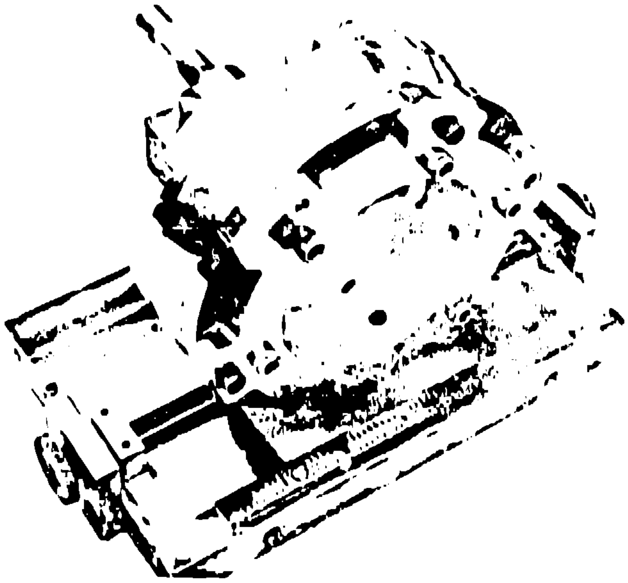

Fig. 6